

537,166

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property  
Organization  
International Bureau



(43) International Publication Date  
17 June 2004 (17.06.2004)

PCT

(10) International Publication Number  
**WO 2004/050979 A1**

(51) International Patent Classification<sup>7</sup>: **D06B 7/02,**  
D06M 11/40

Neville, H. [AU/AU]; 6 Killara Court, Belmont, Victoria 3216 (AU). **RIPPON, John, Anthony** [AU/AU]; 265 Grossmans Road, Torquay, Victoria 3228 (AU).

(21) International Application Number:  
PCT/AU2003/001496

(74) Agent: **GRIFFITH HACK**; 509 St Kilda Road, Melbourne, Victoria 3004 (AU).

(22) International Filing Date:  
13 November 2003 (13.11.2003)

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:  
2002953105 2 December 2002 (02.12.2002) AU

(71) Applicant (*for all designated States except US*): **COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION** [AU/AU]; Limestone Avenue, Campbell, Australian Capital Territory 2612 (AU).

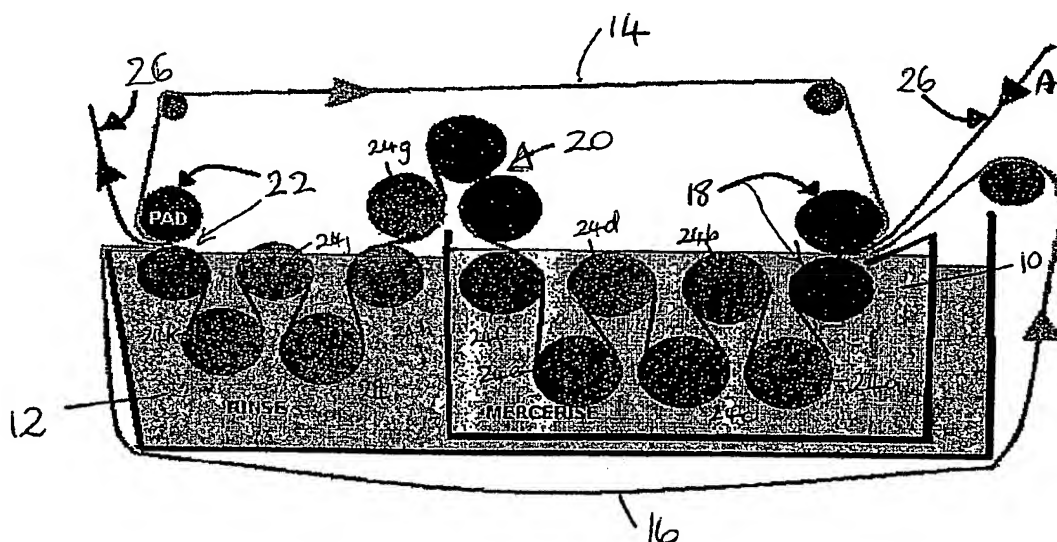
(84) Designated States (*regional*): ARIPO patent (BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

(72) Inventors; and

(75) Inventors/Applicants (*for US only*): **ARNOLD, Lyndon,**

[Continued on next page]

(54) Title: **MERCERISATION OF CELLULOSIC FIBRES**



(57) Abstract: A process for mercerising unconstrained cellulosic fibres comprises transporting the fibres along a transit path through a mercerising zone in which the fibres are contacted with a mercerising liquid, followed by a rinsing zone, in which the fibres are rinsed, wherein the unconstrained cellulosic fibres are held to prevent longitudinal shrinkage during transportation through both zones. The fibres, such as cotton fibres in sliver form, may be held by being pressed between a pair of belts through the zones. Apparatus for conducting the process contains a mercerising zone and a rinsing zone, and a conveyor comprising a pair of surfaces such as belt surfaces for holding the fibres to prevent shrinkage. The belts may be slightly elastic and under tension to slightly stretch the fibres. The belts wind around rollers separated a short distance apart to maintain tension on the belt and on the fibres therebetween.

BEST AVAILABLE COPY

WO 2004/050979 A1

WO 2004/050979 A1



**Published:**

— with international search report

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

MERCERISATION OF CELLULOSIC FIBRESTECHNICAL FIELD

5           The invention relates to a process and apparatus for the mercerisation of unconstrained cellulosic fibres.

DESCRIPTION OF PRIOR ART

10           The beneficial effects obtained by treating cotton with concentrated solutions of various reagents were described by John Mercer in British Patent 13,296 dated 1850. In particular, he noted that treatment of cotton fabrics with sodium or potassium hydroxides,  
15 followed by thorough rinsing, produced shrinkage and thickening similar to that obtained when woollen fabrics are milled. He also noted an increase in tensile strength and improved affinity for dyestuffs following the treatment.

20           Whilst the original process developed by Mercer improved the strength of the cotton fabric, the fabric became thicker and more dense due to shrinkage. The "handle" or lustre of the fabric was also deteriorated as  
25 a consequence of the treatment.

          In 1890 the process now known as mercerising was first described by Lowe in British Patent 4452. Mercerising is a variation on the original process  
30 developed by Mercer under which a cotton fabric or yarn is treated with the mercerising reagent (usually sodium hydroxide) in a stretched condition, followed by rinsing to remove the reagent. Lowe found that shrinkage of the cotton cloth or yarn could be prevented by maintaining  
35 tension in the cotton cloth or yarn during the treatment and rinsing steps. Another important effect found by Lowe was the development of a very high degree of lustre.

- 2 -

Other benefits, previously found by Mercer, such as increased strength and improved affinity for dyes, were also obtained by the method.

5           The mercerisation process, as is now most commonly practised, involves immersing stretched cotton yarn or fabric in a concentrated, aqueous solution of sodium hydroxide (or less commonly another mercerising liquid), followed by rinsing with water and neutralising  
10 any alkali remaining in the rinsed cotton with dilute acid. The powerful swelling action of sodium hydroxide (or other mercerising liquid) has a marked effect on the structure and properties of cellulose. The convolutions present in untreated cotton fibres are removed and their  
15 shape is changed from a flat ribbon-like structure to a smoother more cylindrical form. The effect of the mercerising liquid in swelling the cellulose is that the central cavity (lumen) present in raw cotton largely disappears and the fibre material tends to fill the whole  
20 cross-section. Native cotton contains both crystalline and amorphous cellulose and mercerising increases the proportion of amorphous material in the fibre. Mercerisation also improves the following properties of cotton:

- 25           • Colour yield for a given dye concentration.  
            • Dye coverage of immature fibres.  
            • Chemical reactivity.  
            • Tensile strength.  
            • Strength retention following application of easy-  
30           care finishes.  
            • Lustre.

            It has not been thought possible to obtain the benefits of mercerisation on cotton prior to the assembly  
35 of cotton into yarn or fabric.

- 3 -

Cotton fibres are roughly one inch long, although the fibre length can range from less than half an inch in length to around 2 inches. Until the fibres are aligned and spun into yarn form, assemblages of the fibres, such as carded cotton, slivers and rovings, have not been considered able to be stretched by pulling either end of the assemblage. For example, if either end of a sliver of cotton is pulled, the cotton fibres will slide over one another, and will not individually be stretched. As a consequence, cotton is mercerised in fabric or yarn form.

Mercerisation of cotton in yarn or fabric form has also been found by the applicant to be less complete than formerly believed. Highly concentrated solutions of sodium hydroxide are too viscous at low temperatures to fully wet out all fibres. This is caused by the constraints imposed on the fibres by the yarn twist and the fabric weave. With inadequate penetration of the alkali, only the outer layers of fibres may be mercerised, leaving the fibres inside yarns inadequately treated. Exposed surfaces, especially free fibre ends, are often better treated than fibres buried deep inside yarns. Although superficially mercerised, dark-dyed shades can dye unevenly, and so fade during subsequent laundering, or suffer from "frosting" due to abrasion of the surface.

Accordingly, it is an object of the present invention to provide an alternative process for mercerising cellulosic fibres. Preferably, the process should be capable of being conducted on a continuous basis, and should provide the full benefits of tension mercerisation, including retention of fibre length and development of lustre.

#### 35 BRIEF SUMMARY OF THE INVENTION

The present invention provides a process for

- 4 -

mercerising unconstrained cellulosic fibres comprising:

transporting the unconstrained cellulosic fibres along a transit path through a mercerising zone, in which the unconstrained cellulosic fibres are contacted with a  
5 mercerising liquid followed by a rinsing zone, in which the unconstrained cellulosic fibres are rinsed;

wherein the unconstrained cellulosic fibres are held to prevent longitudinal shrinkage during transportation through both zones.

10

The term "unconstrained" cellulosic fibres as used in this specification refers to oriented or disoriented cellulosic fibres which have not been subjected to spinning or twisting to the extent that they  
15 form a yarn. Accordingly, this term encompasses raw fibres, carded fibres, slivers and rovings, and excludes yarns and fabrics. The cellulosic fibres are suitably in the form of a sliver.

20

The term "cellulosic fibres" has been used in this specification in its broadest sense, and includes cotton fibres, linen fibres, viscose and combinations thereof.

25

The fibres should remain held throughout each of the mercerising and rinsing steps and the passage between these two steps. If the fibres are not held to prevent shrinkage throughout the passage following the mercerising step leading up to the rinsing step, then the fibres will  
30 shrink on release of the holding force. In order to regain the original fibre length it would be necessary to re-stretch the fibres. For this reason, ideally the fibres are held to retain the fibre length during the entire sequence of the mercerising step followed by the rinsing  
35 step, unless the fibres are re-stretched before rinsing. Once most of the mercerising liquid has been removed in the rinsing step, then the holding or tension can be

- 5 -

released, without penalty, for example in an acid neutralising step following the rinsing step.

Preferably, the unconstrained cellulosic fibres are held by being compressed between two surfaces as they travel along the transit path. Indeed, it is preferred that the cellulosic fibres be transported along the transit path between a pair of belts which are pressed together in a plurality of positions through the transit path. The belt should be under tension to maintain a compression force on the fibres to hold them firmly and thus prevent longitudinal shrinkage.

It has been found by the present applicant that excellent results are obtained when the fibres are stretched (placed under stretching tension) during transportation through the mercerising and rinsing zones. The degree of stretching is preferably about 4% or less. Stretching by greater than 4% can reduce fibre strength, and is therefore preferably avoided. Stretching of fibres in the two zones can be achieved by using a slightly elastic belt, which is placed under tension through the mercerising and rinsing zones. Since this arrangement will stretch the fibres in the direction of movement of the belt, it is preferred that the unconstrained cellulosic fibres be oriented. Preferably the unconstrained cellulosic fibres are in the form of carded cotton, a sliver or a roving.

The belts may be made of any suitable material that is resistant to the action of the mercerising liquid. Various polymeric materials are particularly suitable for this purpose, such as polyester. The belts may be of any suitable configuration or construction. For example, the belts may be woven, non-woven, mesh, net-like, perforated, unperforated, or otherwise.

- 6 -

Preferably, the transit path includes nip rollers at the beginning and end of the transit path, which place the belts under tension through the two zones therebetween. The oriented unconstrained cellulosic fibres may be fed into the process between the two belts at the first of the nip rollers. As a consequence of this, if the belts are under tension and are slightly elastic, the fibres are slightly stretched with the belt as they are fed into the process.

10

Preferably the belts pass over guide means at a number of points along the transit path. It has been found by the applicant that for optimum efficiency in preventing slippage of the fibres between the belts, the straight path length between the points at which the belt is in contact with a guide means should be in the range of the average length of the cellulosic fibres or less than this distance. For example, for cotton fibres having an average length of around one inch (2.5 centimetres), the straight path length should be less than 2.8 cm, and preferably 2.5 cm or less.

20

The guide means may be of any suitable construction that fulfills the role of guiding the belts, and therefore this term encompasses surfaces, bars, rollers and so forth. Preferably, the guide means are rollers. The diameter of the rollers is of less significance than the path length between the rollers, however rollers of small diameter are preferred. The diameter of the rollers may advantageously be less than two times the average length of the fibres being treated. More preferably, the diameter of the rollers is less than 1.5 times the average length of the fibres being treated, and most preferably less than or equal to the average length of the fibres.

35

Although the transit path may be of any design

- 7 -

that takes it through the relevant zones, preferably the transit path is a circuitous path that winds around the rollers. More preferably, there are approximately equal numbers of left- and right-hand curves around the rollers in the transit path.

Further nip rollers in addition to those at the beginning and the end of the transit path may be provided. For example, nip rollers may be provided in the region between the mercerising zone and the rinsing zone to squeeze excess mercerising liquid out of the fibres and belts.

The belts may be driven by any appropriate means. For example, the nip roller at the end of the transit path may be configured as a pad mangle which pulls the belt through the transit path.

The mercerising liquid is preferably contained in a mercerising bath, and the rinse liquid in a rinse bath.

Any suitable mercerising liquid may be used in the process of the present invention. However, it is preferred that the mercerising liquid is an alkali. Preferably further, the alkali is a concentrated solution of sodium hydroxide or potassium hydroxide. Appropriate concentrations for the alkali are known in the art. Preferably the concentration of the alkali is between 15 and 30% mass volume (grams per 100 ml), or 35 -55°Tw (Degrees Twaddell - a measure of specific gravity used in the art of the invention). More preferably, the concentration is about 20-22% or about 40 °Tw. Alternative mercerising liquids may include strong acids (eg sulphuric or phosphoric) zinc chloride, calcium chloride and so forth.

Preferably, the mercerising liquid also contains

- 8 -

a wetting agent. Wetting agents are particularly useful for situations where the mercerising liquid is contacted with the fibres at low temperatures and without a preliminary higher temperature stage. Suitable wetting agents (ie ones that are stable in concentrated solutions of alkali) are known in the art.

Preferably, the rinsing liquid is water, however the rinsing liquid may be any other liquid having a pH less than that of the mercerising solution, in the case where the mercerising liquid is an alkali.

The cellulosic fibres may be transported through additional zones after rinsing. For example, the cellulosic fibres may be conveyed through a neutralising zone in which the cellulosic fibres are neutralised with a neutralising liquid. The neutralising liquid may for example be a dilute acid.

According to the present invention, there is also provided an apparatus for mercerising unconstrained cellulosic fibres, the apparatus comprising:

- (i) a mercerising zone;
- (ii) a rinsing zone following the mercerising zone;
- (iii) a conveyor comprising a pair of surfaces for holding the unconstrained cellulosic fibres to prevent longitudinal shrinkage during transportation of the unconstrained cellulosic fibres along a transit path through the mercerising zone and the rinsing zone;
- (iv) driving means for driving the conveyor; and
- (v) pressure means for pressing the surfaces of the conveyor together in the mercerising zone and the rinsing zone to thereby hold the unconstrained cellulosic fibres so as to prevent longitudinal shrinkage of the fibres through the zones.

- 9 -

Preferably, the apparatus also includes a neutralising zone following the rinsing zone. Any other treatment zones may also be incorporated into the apparatus, including as one example, a dyeing zone.

Preferably, the conveyor is configured to enable the unconstrained cellulosic fibres to be held to prevent longitudinal shrinkage throughout the mercerising zone, the rinsing zone and the passage between these two zones. It is not, however, required for the fibres to be held during transportation through the neutralising zone.

Preferably, the conveyor comprises a pair of belts. Preferably the belts are under tension so that, in use, a compression force is maintained on the fibres to hold them firmly and thus prevent longitudinal shrinkage. For the reasons explained above, preferably the belt is slightly elastic so that, in use, an assembly of aligned, unconstrained cellulosic fibres fed between the belts are subjected to a stretching force by the belts in the mercerising and rinsing zones. Other aspects of the belts are described above.

Preferably, the pressure means includes at least two pairs of nip rollers, one pair of nip rollers being located at the end of the mercerising zone, to squeeze out excess mercerising liquid, and the other pair being located at the end of or following the end of the rinsing zone. The pairs of nip rollers should place the belts under tension in the mercerising and rinsing zones therebetween.

Preferably, the straight distance between the points at which the belts come into contact with pressure means ("path length") along the transit path is in the range of the average length of the cellulosic fibres to be

- 10 -

treated in the apparatus, or less than this distance. Preferably, the path length is less than 2.8 cm, more preferably 2.5 cm or less.

5                    Preferably, the pressure means includes a plurality of rollers located along the transit path in the mercerising zone and the rinsing zone. Preferably, the diameter of the rollers is less than two times the average length of the fibres to be treated in the apparatus, more  
10                    preferably less than 1.5 times, and most preferably less than or equal to the average length of the fibres.

                    Preferably, the transit path is a circuitous path that winds around the rollers, and there are approximately  
15                    equal numbers of left- and right-hand curves around the rollers in the transit path.

                    Preferably, the drive means is in the form of a mangle at or downstream of the end of the transit path.  
20                    The drive means may be constituted by the final pair of nip rollers at the end of the transit path which have the dual effect of squeezing out of the fibres.

                    The apparatus may include further components,  
25                    such as further nip rollers to control the transport of the belt through the mercerising liquid and the rinsing and neutralising liquids.

                    Preferably, the mercerising zone includes a  
30                    mercerising bath for containing a mercerising liquid, the rinsing zone includes a rinsing bath for containing a rinsing liquid, and the neutralising zone (if present) includes a neutralising bath for containing a neutralising liquid. The neutralising liquid may for example be a  
35                    dilute acid.

- 11 -

As will be appreciated to persons skilled in the art, the apparatus described above may be utilised for any other processes for treating fibres, including cellulosic and non-cellulosic fibres (including wool), in which it is  
5. desired for the fibres to be held to prevent shrinkage during the treatment. Consequently, the present invention more broadly relates to an apparatus for treating fibres comprising:

- a treatment zone;
- 10 a conveyor comprising a pair of surfaces for holding the fibres to be treated to prevent shrinkage of the material through the treatment zone;
- driving means for driving the conveyor; and
- pressure means for pressing the surfaces of the conveyor  
15 together to hold the fibres to prevent shrinkage of the fibres.

Examples of other treatment zones are zones for subjecting the fibres to dyeing, subjecting the fibres to  
20 alkaline treatments that do not involve mercerising, and so forth.

The other features of the apparatus are as described above.

25

#### BRIEF DESCRIPTION OF THE DRAWINGS

The invention is described further by way of example with reference to the accompanying figure in  
30 which:

Figure 1 is a schematic diagram of the mercerising apparatus in accordance with a preferred embodiment of the invention;

Figure 2 contains scanning electron micrographs  
35 of cross-sections of cotton fibres before and after treatment in the apparatus of Figure 1; and

Figure 3 contains scanning electron micrographs

- 12 -

of side views of the cotton fibres of Figure 2 before and after treatment.

#### DETAILED DESCRIPTION OF THE INVENTION

5

Figure 1 schematically illustrates apparatus according to a preferred embodiment of the invention.

10 The apparatus for mercerising unconstrained cellulosic fibres according to the preferred embodiment as illustrated in Figure 1 includes a first bath 10 containing a mercerising liquid, a second bath 12 containing a rinse liquid, a pair of enveloping conveying belts, including an upper belt 14 having a return loop  
15 passing over the baths, and a lower belt 16 having a return loop passing beneath the baths, and a series of rollers including nip rollers 18, 20, 22 and guide rollers 24a-k for applying a pressing force to press the belts together between rollers 18 and 22. Nip rollers 22 are  
20 constituted as a pad mangle, which drives the belts in the direction indicated by the arrows.

The belts 14,16 are formed from a material (polyester) that is slightly elastic and resistant to the  
25 mercerising liquid in the first bath 10. The belts are approximately 1mm thick, although different thicknesses of belt may be used. The construction of the belts allows the mercerising liquid used in the process to penetrate into the sliver, and also allows the rinsing liquid to  
30 fully rinse the sliver. The belts are driven through a transit path, which extends from nip roller 18 to nip roller 22. The transit path of the belts runs through the first bath 10, out of the first bath and through the second bath 12 in the direction of travel of the belts  
35 14,16.

The transit path of the belts is circuitous and

- 13 -

runs around a series of closely spaced rollers 20 and 24a-k of small diameter. The diameter of the rollers of the preferred embodiment illustrated is 5 cm. This is appropriate for cellulosic fibres of an average length of about 2.5 cm. The diameter of the rollers may be adjusted. The straight path length of the belt between adjacent rollers is measured between the points where it comes into contact with adjacent rollers. The straight path length is approximate to or less than the average fibre length (in this case 2.5 cm). In the preferred embodiment illustrated, the straight path length between adjacent rollers ranges from 2.8 cm to 0 (in the case of the nip rollers). The distance between the rollers may also be adjusted.

The rollers are arranged so that the belt follows a meandering path through the mercerising bath and the rinsing bath, with an approximately equal number of left- and right-hand curves. Of course, other roller arrangements for the rollers are possible.

Unconstrained cellulosic fibres, preferably in the form of one or more slivers 26, are fed between the two belts from nip roller 18 to nip roller 22. The fibres in the sliver are aligned to the direction of travel of the belts and the direction of feeding of the sliver into the apparatus (arrow A).

The pad mangle 22 pulls the belt along the transit path and applies tension to the belt between the pad mangle and nip roller 18. As the belt is slightly elastic, the belt is stretched commencing at nip rollers 18. The input sliver 26 that is fed between the belts at nip rollers 18 is compressed between the belts to be held sufficiently firmly to prevent shrinkage of the fibres as they pass through the transit path. The pressing of the sliver causes it to spread a little. If there are several

- 14 -

slivers fed into the apparatus across the width of the belts, the slivers are suitably spaced adjacent one another in a single layer. With spreading of the slivers, there may be a little overlap between the pressed-out edges of the adjacent slivers.

Since the belt is slightly stretched as it commences the transit path at nip rollers 18, so too are the fibres between the belts. The degree of stretching of the fibres is around 1-2%. The fibres remain in the stretched state as they travel along the transit path through the mercerising bath, out of the mercerising bath and through the rinsing bath. The tension applied to the fibres also assists to maintain the fibres parallel to the direction of travel through the apparatus. In the embodiment illustrated, the fibre load of cotton fed into the apparatus is such that the belt separation (in this case, the distance between the belts, not including the width of the belts themselves) is up to about 2-3 mm. This corresponds to 5 kTex thick cotton slivers being fed into the apparatus. Cotton slivers of this size spread a little to be approximately 10-15mm wide, and therefore an arrangement of three 5 kTex slivers will cover a belt width of about 30-40mm. Thicker slivers of cotton or other fibres may be fed into the apparatus up to 15kTex per 30mm of belt width.

As explained previously, the small diameter of the rollers 20 and 24a-k and the short path length between the rollers (relative to the fibre length) has been found to provide optimum efficiency in preventing slippage of the fibres between the facing surfaces of the pair of belts 14,16.

Nip rollers 20 are positioned above the mercerising bath and squeeze excess mercerising liquid out of the sliver and belts. The excess mercerising liquid

- 15 -

drains back into the bath.

In the preferred embodiment of the invention, the mercerising liquid is an aqueous solution of sodium hydroxide. The concentration of the sodium hydroxide is 20-22% mass volume or 40°Tw at 15°C, which corresponds to a specific gravity of 1.2 or approximately 21 grams of sodium hydroxide per 100 ml of water. The mercerising liquid also contains a wetting agent in the amount of 5 g/L to 25 g/L. One of the wetting agents which is stable in strong alkali known in the art is used in the preferred embodiment. Wetting agents are useful in the situation where the mercerising liquid is contacted with the fibres at low temperatures. If the process was modified to bring the mercerising liquid into contact with the fibres at a higher temperature, prior to reducing the temperature of the mercerising liquid contacting the fibres to the mercerising treatment temperature, then a wetting agent may be dispensed with.

20

The mercerising solution is maintained at a temperature in the range of -5°C to 25°C. The temperature is suitably towards the lower end of this range.

25

The rinsing liquid has a pH less than the pH of the mercerising solution. Water is used in the preferred embodiment.

30

The sliver 26 may also be transported through additional zones (not shown) to expose the sliver to further solutions. An additional zone may include a solution containing a dilute acid to further neutralise any remaining mercerising liquid in the sliver and/or belts.

35

Whilst baths are used for containing the mercerising liquid and rinsing liquid in the preferred

- 16 -

embodiment illustrated, it will be appreciated to a person skilled in the art of the invention that a chamber or chambers in which the unconstrained cellulosic fibres are sprayed with the liquids could be used in place of the  
5 baths. The term "zone" should accordingly be interpreted broadly to encompass this.

The speed at which the sliver 26 is transported through the mercerising bath 10 (and the length of the  
10 bath) should be sufficient to ensure that the mercerising liquid will have fully penetrated the sliver and so that the mercerisation of the fibre has been effected. This also applies to the other baths through which the sliver is conveyed. For related reasons, the number of rollers  
15 over or through which the fibres progress should be sufficient for the fibres to remain in contact with the mercerising liquid for enough time for full penetration and mercerising to be effected.

20 Although the cellulosic fibres are held in a compressed condition between the tensioned belts 14,16, it has been surprisingly found by the present applicants that the fibres are still able to swell and expand in diameter. Consequently, the fibres are changed by the mercerisation  
25 process from a flat, ribbon-like shape that is typical of untreated cellulosic fibres to the more circular cross-section that is characteristic of mercerised cellulosic fibres.

30

### EXAMPLES

#### Example 1

35 A 5 ktex sliver, composed of cotton fibres, was treated in the apparatus shown in Figure 1. The sliver was fed between two belts into a pair of nip rollers. It was

- 17 -

carried between the belts around a series of rollers immersed in a tank containing the mercerising liquor, which consisted of an aqueous solution of sodium hydroxide at a concentration of 21.4% (mass/volume), maintained at a temperature below 10°C. The treatment liquor also contained 7g/L of a wetting agent (Leophen MC [BASF]). The speed of the belt and sliver was set to give an immersion time in the sodium hydroxide solution of 40 seconds. After squeezing out the excess sodium hydroxide solution, the sliver, while still held under tension, was then passed through a rinse bath, containing water, for 30 seconds. After rinsing, excess liquid was removed with a pair of squeeze rollers (not shown in Figure 1). The sliver was then passed into a bath containing dilute acetic acid, maintained at pH 5 (not shown in Figure 1), where the remaining alkali was neutralised.

A control was prepared by treating cotton sliver with the solution of sodium hydroxide and wetting agent, described above; but in this case, the material was allowed to fully relax in the treatment solution and during the rinsing and neutralisation steps (this sample is referred to as slack mercerised).

Examination of the slivers after drying showed that the sample mercerised under tension on the Belt Mercerising Machine was more lustrous than the untreated material and also more lustrous than the slack mercerised sample. The lustre of the sliver was twice that of the untreated material. The fibre length, strength and elongation at break of the untreated and treated slivers were measured. The data in Table 1 show that the slack mercerised fibres contracted in length, whereas the fibres mercerised under tension were slightly longer than the untreated material. Both the slack mercerised and belt mercerised fibres were stronger and had a higher elongation at break than the untreated cotton.

**Table 1:**

**Physical Properties of Untreated, Slack Mercerised and Belt (Tension)**

**5 Mercerised Cotton**

	Mean fibre length (inches)	Fibre Strength (grams force per tex) (a)	Elongation at Break (%)
Untreated	1.17	32.5	6.0
Slack Mercerised	1.12 (- 4.3 %)	42.3 (+ 30.2%)	13.3
Belt Mercerised (under tension)	1.18 (+ 0.9 %)	41.5 (+ 27.7%)	8.9

The figures in brackets show percentage change compared with untreated cotton, gauge length 1/8 inch - see middle  
10 column above.

Scanning electron micrographs of untreated and belt mercerised cotton (Figures 2 and 3) show that the belt mercerising technique successfully changed the fibre  
15 cross-section from the kidney-shaped, ribbon-like cross-section of normal cotton fibres to the more circular shape characteristic of mercerised cotton. Figure 3 shows that the mercerised fibre surfaces are smoother, less contorted and more cylindrical, so they will show enhanced lustre.

20

**Example 2**

Example 1 was repeated using 5 ktex slivers of two other samples of cotton fibres. The results were compared to  
25 control examples, one of which was unmercerised, and the second of which was slack mercerised. The composition and

concentration of the solutions were as outlined in Example 1.

The properties of the fibres were tested and the results outlined in Table 2. The results in Table 2 incorporate those outlined in Table 1.

For a given sample of cotton, the tenacity-at-break for the "belt"- (held) and slack-mercerised slivers were similar, but significantly higher than their un-mercerised Controls. The elongations-at-break for the belt-mercerised samples were similar to their Controls, whereas the slack-mercerised slivers showed significant increases in elongation - a further confirmation of the effects of the high fibre shrinkage that occurs when fibre is unconstrained during treatment.

Measurements also showed that the sliver of Sample 2 was 2.5 times more lustrous after the belt-mercerisation process than its un-mercerised Control.

Table 2:

25      Fibre length, strength and fineness for three mercerised  
cotton samples comparing Controls with "belt"-mercerised,  
or mercerised without length constraint (slack).

Sample & Mercerisation	Upper Half Mean Length	Fibre Shrinkage	Short Fibre Index (% < 0.5 inch)	Length Uniformity (%)	Breaking Tenacity (gf/tex)	Elongation To Break (%)	Micronaire $\mu\text{g/inch}$
Sample 1 COTTON SLIVER							
Un-mercerised	1.19	0	2.6	84.3	36.0	5.8	4.3
Belt-mercerised	1.24	-4.2	1.1	84.6	47.5	5.6	4.6
Sample 2 COTTON SLIVER							

- 20 -

Un-mercerised	1.17	0	3.6	82.5	32.5	6.0	4.2
Belt-mercerised	1.18	-0.9	4.5	82.0	41.5	8.9	4.8
Slack mercerised	1.12	5.1	5.6	80.6	42.3	13.3	5.3
Sample 3 COTTON SLIVER							
Un-mercerised	1.18	0	3.0	83.5	35.9	6.1	4.2
Belt-mercerised	1.16	1.3	4.1	82.5	42.5	7.1	4.8
Slack mercerised	1.09	6.0	5.0	81.3	45.3	16.3	5.6

(Note: a positive figure under shrinkage refers to shrinkage; negative refers to extension - negative being sought)

### Spinning and Dyeing results

5

Samples of "belt" (held)-mercerised and control slivers (Sample 2 cotton) were successfully drawn down from 5 ktex to 1 ktex, and then spun into 40 tex singles yarns at 700 tpm. Two-folding at 400 tpm was also been successfully completed. At no time was it necessary to add lubricants during the drawing and spinning processes. This confirms that the mercerising process of the examples is not severe enough to remove the natural wax coating from the fibres.

10

The belt-mercerised and control yarns were simultaneously cone dyed in the same bath and knitted into small swatches. Examination of both the yarns and the knitted samples confirmed an increase in lustre and a greater depth of shade for the belt-mercerised yarn.

20

The process described above gives rise to the following benefits:

25

- Improved evenness of treatment resulting from subsequent processing carried out after mercerising the fibre.
- Increased fibre strength, compared with normal cotton. This leads to improvements in spinning

- 21 -

performance, in particular the possibility of higher spinning frame speeds.

- 5           •   Possibility of blending mercerised cotton fibre with alkali-sensitive materials, in particular keratin fibres such as wool.
- 10          •   Knitted fabrics can be made from mercerised cotton. Although knitted fabrics can be mercerised, the mercerisation process tends to give a decrease in fabric bulk. This problem is avoided when cotton is mercerised as loose fibre or sliver.

15           The above preferred embodiment and example are provided by way of illustration of the inventive concept only. Various modifications may be made to the preferred embodiment and example without departing from the spirit and scope of the invention.

**CLAIMS:**

1. A process for mercerising unconstrained  
cellulosic fibres comprising:
  - 5 transporting the unconstrained cellulosic fibres  
along a transit path through a mercerising zone, in which  
the unconstrained cellulosic fibres are contacted with a  
mercerising liquid followed by a rinsing zone, in which  
the unconstrained cellulosic fibres are rinsed;  
10 wherein the unconstrained cellulosic fibres are  
held to prevent longitudinal shrinkage during  
transportation through both zones.
2. The process of claim 1, wherein the  
15 unconstrained cellulosic fibres are raw fibres, carded  
fibres, slivers or rovings.
3. The process of claim 1, wherein the  
unconstrained cellulosic fibres are in the form of a  
20 sliver.
4. The process of any one of claims 1 to 3, wherein  
the fibres are held to retain the fibre length during the  
entire sequence of the mercerising step followed by the  
25 rinsing step, including the passage between the two steps.
5. The process of any one of claims 1 to 4, wherein  
the unconstrained cellulosic fibres are held by being  
compressed between two surfaces as they travel along the  
30 transit path.
6. The process of claim 5, wherein the cellulosic  
fibres are transported along the transit path between a  
pair of belts which are pressed together in a plurality of  
35 positions through the transit path.

- 23 -

7. The process of any one of claims 5 and 6, wherein the fibres are stretched during transportation through the mercerising and rinsing zones.

5 8. The process of claim 7, wherein the degree of stretching is about 4% or less.

9. The process of any one of claims 7 and 8, wherein the belt is slightly elastic and placed under  
10 tension through the mercerising and rinsing zones.

10. The process of any one of claims 7 to 9, wherein the unconstrained cellulosic fibres are oriented.

15 11. The process of any one of claims 6 to 10, wherein the transit path includes nip rollers at the beginning and end of the transit path, which place the belts under tension through the two zones therebetween.

20 12. The process of any one of claims 6 to 11, wherein the belts pass over guide means at a number of points along the transit path.

25 13. The process of claim 12, wherein the straight path length between the points at which the belt is in contact with a guide means is in the range of the average length of the cellulosic fibres or less than this distance.

30 14. The process of claim 13, wherein the straight path length is less than 2.8 cm.

15. The process of any one of claims 12 to 14, wherein the guide means are rollers.

35

16. The process of claim 15, wherein the diameter of the rollers is less than two times the average length of

- 24 -

the fibres being treated.

17. The process of claim 15, wherein the diameter of the rollers is less than 1.5 times the average length of the fibres being treated.

18. The process of any one of claims 15 to 17, wherein the transit path is a circuitous path that winds around the rollers.

19. The process of any one of claims 12 to 18, wherein nip rollers are provided in the region between the mercerising zone and the rinsing zone to squeeze excess mercerising liquid out of the fibres and belts.

20. The process of any one of claims 6 to 19, wherein the belts are driven by a pad mangle at the end of the transit path which pulls the belt through the transit path.

21. The process of any one of claims 1 to 20, wherein the cellulosic fibres are transported through additional zones after rinsing.

22. The process of claim 21, wherein the cellulosic fibres are conveyed through a neutralising zone in which the cellulosic fibres are neutralised with a neutralising liquid.

23. An apparatus for mercerising unconstrained cellulosic fibres, the apparatus comprising:

- (i) a mercerising zone;
- (ii) a rinsing zone following the mercerising zone;
- (iii) a conveyor comprising a pair of surfaces for holding the unconstrained cellulosic fibres to prevent longitudinal shrinkage during transportation of the unconstrained cellulosic

- 25 -

fibres along a transit path through the mercerising zone and the rinsing zone;

- (iv) driving means for driving the conveyor; and  
(v) pressure means for pressing the surfaces of the conveyor together in the mercerising zone and the rinsing zone to thereby hold the unconstrained cellulosic fibres so as to prevent longitudinal shrinkage of the fibres through the zones.

24. The apparatus of claim 23, wherein the apparatus includes a neutralising zone following the rinsing zone.

25. The apparatus of claim 23 or claim 24, wherein the conveyor is configured to enable the unconstrained cellulosic fibres to be held to prevent longitudinal shrinkage throughout the mercerising zone, the rinsing zone and the passage between these two zones.

26. The apparatus of any one of claims 23 to 25, wherein the conveyor comprises a pair of belts.

27. The apparatus of claim 26, wherein the belts are under tension so that, in use, a compression force is maintained on the fibres to hold them firmly and thus prevent longitudinal shrinkage.

28. The apparatus of claim 27, wherein the belt is slightly elastic so that, in use, an assembly of aligned, unconstrained cellulosic fibres fed between the belts are subjected to a stretching force by the belts in the mercerising and rinsing zones.

29. The apparatus of any one of claims 26 to 28, wherein the pressure means includes at least two pairs of nip rollers, one pair of nip rollers being located at the end of the mercerising zone, to squeeze out excess

- 26 -

mercerising liquid, and the other pair being located at the end of or following the end of the rinsing zone.

30. The apparatus of any one of claims 26 to 29,  
5 wherein the straight distance between the points at which the belts come into contact with pressure means ("path length") along the transit path is in the range of the average length of the cellulosic fibres to be treated in the apparatus, or less than this distance.
- 10 31. The apparatus according to claim 30, wherein the path length is less than 2.8 cm.
32. The apparatus of any one of claims 26 to 31,  
15 wherein the pressure means includes a plurality of rollers located along the transit path in the mercerising zone and the rinsing zone.
33. The apparatus of claim 32, wherein the  
20 diameter of the rollers is less than two times the average length of the fibres to be treated in the apparatus.
34. The apparatus of claim 32 or 33, wherein the  
25 transit path is a circuitous path that winds around the rollers, and there are approximately equal numbers of left- and right-hand curves around the rollers in the transit path.
35. The apparatus of any one of claims 26 to 34,  
30 wherein the drive means is in the form of a mangle at or downstream of the end of the transit path.
36. The apparatus of any one of claims 23 to 35,  
35 wherein the mercerising zone includes a mercerising bath for containing a mercerising liquid, the rinsing zone including a rinsing bath for containing a rinsing liquid.

37. An apparatus for treating fibres, comprising:  
a treatment zone;  
a conveyor comprising a pair of surfaces for holding the  
5 fibres to be treated to prevent shrinkage of the material  
through the treatment zone;  
driving means for driving the conveyor; and  
pressure means for pressing the surfaces of the conveyor  
together to hold the fibres to prevent shrinkage of the  
10 fibres.

38. Product produced by the process of any of claims  
1 to 22 or in the apparatus of any one of claims 23 to 37.





Untreated



Belt Mercerised

Figure 2

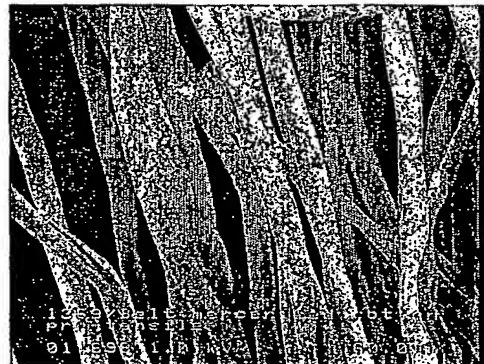


Figure 3

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/AU2003/001496

## A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl. <sup>7</sup>: D06B 7/02, D06M 11/40

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

DWPI: D06B 7/02, D06M and keywords: mercer, sliver and similar terms

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 2236770 A (LEUNG SUI KI) 17 April 1991 Whole document	1-5, 21, 22, 38
A	GB 2125836 A (TOYO BOSEKI) 14 March 1984 Page 2 lines 102-106	
A	Patent Abstracts of Japan JP 08-035164 A (TOSUKO KK) 6 February 1996	

☐

Further documents are listed in the continuation of Box C

☒

See patent family annex

<p>* Special categories of cited documents:</p>	
"A" Document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E" Earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"L" Document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"O" Document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family
"P" Document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search  
25 November 2003

Date of mailing of the international search report  
9 JAN 2004

Name and mailing address of the ISA/AU  
AUSTRALIAN PATENT OFFICE  
PO BOX 200, WODEN ACT 2606, AUSTRALIA  
E-mail address: pct@ipaustalia.gov.au  
Facsimile No. (02) 6285 3929

Authorized officer  
  
M. BREMERS  
Telephone No : (02) 6283 2052

# INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU2003/001496

## Box I Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos :  
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos :  
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. ☐ Claims Nos :  
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a)

## Box II Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

See Extra Sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☒ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.: 1-36 and 38

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.  
☐ No protest accompanied the payment of additional search fees.

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU2003/001496

### Supplemental Box

(To be used when the space in any of Boxes I to VIII is not sufficient)

#### Continuation of Box No: II

The international application does not comply with the requirements of unity of invention because it does not relate to one invention or to a group of inventions so linked as to form a single general inventive concept. In coming to this conclusion the International Searching Authority has found that there are different inventions as follows:

1. Claims 1-36 are directed to a process and apparatus for the mercerisation of unconstrained cellulosic fibres wherein the fibres are held to prevent shrinkage during transport through the mercerising and rinsing zones. It is considered that the prevention of shrinkage of unconstrained fibres during mercerisation comprises a first "special technical feature".
2. Claim 37 is directed to an apparatus for the prevention of shrinkage of fibres during a treatment. It is considered that the conveyor for holding the fibres comprises a second "special technical feature".

These groups are not so linked as to form a single general inventive concept, that is, they do not have any common inventive features, which define a contribution over the prior art. The common concept linking together these groups of claims is the prevention of fibre shrinkage during a treatment. However this concept is not novel in the light of the prior art described at page 1 lines 33-36 of the present application and GB 2236770 A (LEUNG SUI KI) 17 April 1991, see the abstract. Therefore these claims lack unity a posteriori.

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/AU2003/001496

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member
GB	2236770	NONE
GB	2125836	NONE
JP	8035164	NONE
		END OF ANNEX